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STUDIES OF THICKENED LIQUIDS

AND MISCELLANEOUS FLAME THROWER PROBLEMS

by
E. E. Bauer and E. K. Carver
Eastman Kodak Company

Report OSRD No. 6236
Copy No. 35
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STUDIES OF THICKENED LIQUIDS AND MISCELLANEOUS FLAME THROWER PROBLEMS

Service Directives: CWS-10, 12, 21

Endorsement (1) Professor H. C. Hottel, Chief, Section 11.1 to Dr. H. M. Chadwell, Chief, Division 11.

Forwarding report and noting:

"The attached report summarizes 3-1/2 years of activity of an Eastman Kodak group in many fields covered by the general title, "Studies of Thickened Liquids." Starting as a fundamental study of the rheology of thickened fuels the project moved on to include a wide range of problems associated with development work by other contractors on incendiaries and flame throwers. The work included studies of small jet performance, the effect of oxidation and of moisture on the stability of Napalm, Napalm manufacturing problems, dehydrating agents, the effect of various peptizers on thickened fuels, anomalous effects of compounding temperature and of storage temperature on the consistency of Napalm, various fiber additives, the appraisal of other thickening agents than Napalm, the performance of portable flame throwers with various thickeners, the pumping characteristics of Napalm thickening fuels, and methods of mixing Napalm and gasoline. The last two fields of activity led to development of equipment for mixing Napalm under subcontract with the Ferro Enamel Corp. and a purchase order on the Cleaver-Brooks Co., and to the development by the Eastman group itself of a pump-operated flame thrower. At the end of the war this principle of mechanized flame thrower design held great promise, and the work is continuing under a direct contract with the Chemical Warfare Service."

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OSRD No. 6236

Endorsement (2) from Dr. H. M. Chadwell, Chief, Division 11 to Mr. Cleveland Norcross, Executive Secretary of the National Defense Research Committee.

Forwarding report and noncurring.

This is a final report under Contract 11-300, OEMsr-538 with the Eastman Kodak Company.

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EASTMAN KODAK COMPANY
DEPARTMENT OF MANUFACTURING EXPERIMENTS
Rochester, N. Y.

File No. 11-300
Contract OEMsr-538
Problems CWS 10, 12, 21

Final Report to Division 11 of the National Defense Research Committee on "STUDIES OF THICKENED LIQUIDS"

October 23, 1945

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Rheological Studies.

The work under contract OEMsr-538 was undertaken in April 1942 with a view to determining the rheological properties of thickened vesicants and the visco-elastic fluids then being developed for use as fuels in flame throwers and incendiary bombs. These properties were studied by means of Clark-Hodsman, MacMichael, and High Pressure Capillary viscosimeters, a special Jeweler's Lathe viscosimeter which was developed for use in the study of short time effects and a Resonance Elastometer with the aid of which rigidity and viscosity could be determined at frequencies up to seventy cycles per second.

The thickened hydrocarbon fluids which were studied included Napalm, then known as X-104, other aluminum soap thickened fuels developed by the Columbia University and Massachusetts Institute of Technology Laboratories of the Chemical Warfare Service, isobutyl methacrylate interpolymer gels and other isobutyl methacrylate formulations, such as F-241, 2765 and 2769, developed by DuPont. All these fluids were found to be pseudoplastic, having much higher viscosities at low than at high rates of shear. In addition, all showed measurable rigidity, and some displayed thixotropy, yield value, work hardening, and after-elastic effects in varying degrees.

Unignited 1/8-inch jet experiments were made in an attempt to correlate performance of thickened fluids in the flame thrower (i.e. range) with their rheological properties. No significant correlation was observed between range and any variable except the apparent viscosity of the fluid. When viscosity at a rate of shear of four reciprocal seconds (determined with MacMichael) was plotted against range for a number of fluids compared at the same initial (nozzle) kinetic energy, the data for Newtonian, pseudoplastic, thixotropic and dilatant fluids covering a viscosity range of 0.1 to 8000 poises was found to fall on the same line. The only significant deviations were for liquids of low viscosity and high surface tension. It was thus concluded that the primary rheological requirement for flame thrower fuels is pseudoplasticity, the low apparent viscosity at high rates of shear permitting high nozzle velocity at reasonable pressures and the high apparent viscosity at low rates of shear preventing breakup of the jet by the drag of the surrounding air. However, certain liquids which are short (i.e. lack stringiness) and which also have high moduli of rigidity were found to show greater breakup in the air and less range than stringy liquids of the same apparent viscosity. An extensiometer was developed for the evaluation of stringiness of these fuels and some evidence was found of a minimum extensibility required for satisfactory performance, any increase above this value, however, having no beneficial effect (January 1943).

As a result of the fundamental study of the rheological properties of incendiary gels, it was concluded that empirical tests involving extensibility and breakup and adhesion on impact, in conjunction with a determination of apparent viscosity at the appropriate rate of shear, would be of more value in evaluating the fuels than a thorough study of the complex rheological properties. Details of the rheological studies of the incendiary gels will be found in progress reports of June 1942 to March 1943, OSRD No. 1589 and OSRD No. 1893.

Rheological measurements were also carried out on vesicants, both thickened and unthickened, using MacMichael and Ostwald-Cannon-Fenske (capillary) viscosimeters and a resonance elastometer modified for use with fluids of low viscosity. Mustard, and mixturds of mustard and Lewisite thickened with polystyrene, methyl methacrylate, isobutyl methacrylate, gelva, alloprene, rubber and tornesit(chlorinated rubber) were studied. Most of the samples exhibited pseudoplasticity in varying degrees, and a few, particularly those of highest molecular weight, had measurable elasticity. However, no relationship was found between the rheological properties and the drop size distributions on the DuPont mortar tests. (August 1942, April-July 1943)

Stability Studies.

In conjunction with the rheology studies, the stability of the various incendiary gels was determined over a wide range of temperatures. This was done by means of sealing the material in a glass tube along with a steel ball of appropriate size and measuring the rate of fall of the ball from time to time. Some of the fuels were stored in mason jars containing steel, bonderized steel, etc., the material being removed for viscometric measurements at the end of the keeping period. The excellent stability displayed by the Napalm type fuels at all temperatures played a large part in the decision to produce this thickener in quantity. The manufacture of Napalm was begun by Nuodex in December 1942 (July 1942 - January 1943).

Specification Viscosimeter.

Extensive tests were carried out in conjunction with the Standard Oil Development group and the Chemical Warfare Service to determine the best viscosimeter for use in specification testing of Napalm. A modified (paddle wheel) Stormer, a Plumb-Bob viscosimeter and the Gardner Mobilometer were compared with a number of batches of Napalm and it was concluded that the Gardner instrument was the most consistent, reliable and fool-proof; hence, it was adopted and was used throughout the war in the testing of Napalm.

Moisture and Oxidation.

During the work on rheological properties of Napalm fuels, poor agreement was often attained between workers using what were supposed to be identical samples of Napalm. An in-

vestigation of these anomalies led to the conclusion that they were due to two main causes: (a) oxidation of the solid soap during or after drying, (b) variable moisture content due to gain or loss of water during storage. A study was made of these phenomena by means of drying samples of wet pulp at various temperatures and conditioning dried samples of the soap at various relative humidities.

The effect of moisture content on viscosity, extensibility and gelation rate of several Napalms was studied* and various methods of determining moisture content, including vacuum oven, benzene distillation, and Karl Fisher method, were investigated. It was concluded that the benzene distillation method was most suitable for use in production control and a variation of this method was adopted for specification use.

Oxidation, which often occurred during drying or on subsequent storage of the soap, was found to be associated with reduction in iodine number and to be promoted by iron and manganese which occurred as impurities in the Victory grade alum. Extensive studies were made of the oxidation susceptibility of Napalms by means of the Mackey test, in which the soap is exposed at elevated temperature to a current of air, and the peroxide value test (January-March, September-December 1943).

Napalm Manufacture.

The problems associated with moisture and oxidation and various other phases of the general problem of producing Napalm reliably in large quantities were studied by this group in cooperation with other N.D.R.C. personnel, Chemical Warfare Service and the Napalm manufacturers. Results of these studies along with a detailed discussion of a preferred manufacturing procedure are given in O.S.R.D. No. 2036 and 2036A.

Surveillance.

In the fall of 1943, sample M69 incendiary bombs filled with Napalm were regularly received from each of the filling plants along with samples of the soaps from which the fuels were prepared. The stability of the fuel in the bombs was followed by means of an oscillimeter which gave a measure of viscosity without requiring the bombs to be opened. The thickening power and oxidation susceptibility of the accompanying soap samples were determined. As a result of this study it was concluded that the thickening power and oxidation resistance of the Napalm soaps and the stability of the fuels in the bombs were quite satisfactory (September 1943 - March 1944).

* "Effect of Moisture on X-104 Soaps", E.E.Bauer and E.K.Carver, November 1942.

Late in 1943 one-hundred pound samples of Napalm, representative of regular production, were received from each of the ten manufacturers. Stability of gels made from these Napalms at varying moisture contents and concentrations were studied at ordinary and elevated temperatures. Results of this investigation which was carried out in conjunction with Standard Oil Development Company are given in O.S.R.D. No. 3508. As a result of the wide variation in consistencies of these fuels at low concentrations, it was recommended that the effect of this variation on flame thrower performance be studied.

Dehydrating Agents.

Incorporation of moderately strong dehydrating agents such as silica gel, calcium chloride and magnesium sulfate in Napalm fuels was found to increase their consistency and stability. Silica gel was found to be the most efficient agent in this respect (February 1944). Tests with Napalm samples from a number of manufacturers, however, showed that silica gel does not appreciably increase uniformity of consistency among the various Napalms and that the increase in stability is no greater than would result from increasing Napalm concentration to give the same consistency (July 1945). However, fuels containing excess silica gel showed considerably greater resistance to the deleterious effect of xylenol, amines, acid soldering flux, potassium acetate, etc., than regular Napalms of equal consistencies.

Flame Thrower Studies.

Early in 1944 two M1A1 flame throwers were obtained from Chemical Warfare Service and these were fired with Napalm fuels of various concentrations to determine the effect of consistency on flame diameter and range. The difference in flame thrower performance between Nuodex and Imperial Napalms of equal concentrations was pointed out and as a result, the Imperial soap was standardized for packaging in the 5-1/4 pound containers for field mixing of fuels. A Kodachrome movie showing effect of fuel consistency on flame thrower performance along with a demonstration of mixing of Napalm fuels was produced and was circulated widely in both the European and Pacific theaters of war (May-July 1944).

Flame thrower performance of fuels compounded with xylenol and other peptizers (materials which lower consistency), incorporated to improve pourability and stability, was determined. As a result of extensive tests with the M1A1 (June 1944), E5R1 (November 1944) and M2-2 (August 1945) flame throwers, it was concluded that Gardner consistency, irrespective of Napalm concentration, is a reliable guide to performance of the flame thrower with Napalm fuels. Thus, additives which impart properties which are desirable in the preparation, handling or storage of fuels may be incorperated even though they raise or lower the consistency so long as the Napalm concentration is adjusted to give the desired consistency.

Considerable research was carried out as a result of the Infantry Board's request for a fuel which would combine the principle advantages of unthickened and thickened fuels (i.e. fierceness of burning and long range, respectively) (July and August 1944). Fuels of about 25 grams Gardner consistency were found to most nearly give the desired flame characteristics. Because 2-1/2 to 3% regular Napalm fuels which have about this consistency have such long stir times and poor stability, it was decided that 4.2% Napalm reduced to 25 grams Gardner with xylenol or water was the best solution to the problem. In connection with this study, a comparison was made of several peptizers, including the alcohols, catechol, and turkey red oil, in addition to water and xylenol.

M1A1 Y and Ball Valves.

The performance of the M1A1 flame thrower was found to depend considerably upon which valve was used in the flame gun. With the Y valve, breakup is considerably greater than with the ball valve and the range is considerably less. With 4.2% Napalm, range was found to be 65 yards with the ball valve and 45 yards with the Y valve (February 1945).

"Minimum Consistency" Fuels.

It was found that when the amount of water in a Napalm fuel is increased beyond approximately one-tenth the soap content, no appreciable further decrease in consistency takes place. These "minimum consistency" fuels have the obvious advantage that they are not affected by small additional quantities of water as are regular Napalm fuels. Fuels of this type containing 6.3% Napalm and 3.5% water were prepared from representative Napalms from several manufacturers. They were found to have consistencies of 15 to 20 grams and were quite stable at ordinary and elevated temperatures. Their flame thrower performance is similar to that of regular Napalms of the same consistency (March and April 1945).

Temperature Effects.

The temperature coefficient of viscosity of unpeptized Napalm fuels is small compared with that of ordinary (Newtonian) liquids. This was observed early in the study of rheological properties, and was attributed to the decreased rate of "healing" (structure formation) at low temperatures, this effect almost counteracting the normal increase in viscosity with decrease in temperature (O.S.R.D. No. 1893, p. 17). Napalm fuels peptized with xylenol have much higher consistencies at low temperatures than at high, and those peptized with water decrease in consistency with decrease in temperature. By using the optimum proportions of xylenol and water it is possible to produce a fuel whose viscosity is almost independent of temperature (December 1944). The best single peptizers from the standpoint of low temperature coefficient of viscosity are the lower aliphatic alcohols (July, August 1945).

When Napalm fuels are mixed and stored at 50-60°F (approximately the minimum temperature at which Napalm can be dispersed without a peptizer), their consistencies are very much higher than when the fuel is mixed at 80° F. For example, a 2% gel may have a consistency of 80 grams instead of 10 grams, a 4% may have a consistency of 250 instead of 100 grams. The difference in flame thrower performance between these abnormal fuels and ordinary fuels of the same Napalm concentration is not as great as would be expected on the basis of consistency, this being the most striking exception to the rule that Gardner consistency irrespective of concentration determines performance (December 1944, March 1945).

Most Napalm soaps increase considerably in thickening power on storage at elevated temperatures in hermetically sealed containers. Thus, 4% Imperial Napalm may have a consistency of 150-200 grams if the soap has been stored for two weeks at 160°F, as compared to a normal consistency of 75 grams. The increase in consistency is accompanied by increase in moisture content and decrease in extractable acid, indicating that some of the uncombined acid has combined with alumina or mono-soap, water being produced by the reaction. Storage at 120° F. was found to have comparatively little effect on thickening power in three months, indicating that little difficulty would be encountered under ordinary storage conditions (February-June 1945).

Field Viscosimeter.

Extensive tests were carried out in an attempt to develop a viscosimeter for field evaluation of thickened fuels (December 1944 to February 1945). A simple efflux viscosimeter which could be fabricated from materials available in the field was devised. This consists of a C ration can with a 1/4 or 1/2 inch circular opening in the bottom. This viscosimeter is suitable for fuels with consistencies up to 250 grams Gardner. The C.W.S. Ball Viscosimeter is capable of covering the consistency range up to 500 grams Gardner if balls up to 9/16-inch diameter and a 100 second fall time are employed. The ball viscosimeter gives somewhat better agreement with the Gardner than does the efflux viscosimeter. Extensive tests were also made with a grease gun viscosimeter.

Mechanized Mobilometer.

A device for mechanically forcing the Gardner plunger through the sample in the tube at a rate of 0.1 cm./sec. and reading the resulting force with a spring scale was developed. By employing four or more mobilometers with a single driving mechanism and using scales with maximum indicating pointers, a considerable saving of time can be effected in the routine testing of Napalm, about 16 to 20 tests per hour being possible. The instrument was tested at the Imperial Paper and Color Corp. plant and was found to give as reliable results as the manual method of operation (March-July 1945, Formal Report to be issued).

Field Mixing Unit.

Preliminary tests were carried out on a method of mixing Napalm by recirculating the gasoline-Napalm mixture in a drum with a small pump driven by a gasoline engine. This principle was later embodied in the field mixing unit (E-11) developed by Cleaver-Brooks (October 1944).

Navy Mixer.

In the fall of 1944 the Navy requested the development of a continuous mixing device for use on the decks of aircraft carriers. Tests were carried out with a Premier Colloid Mill and a Jesperson Mixer, but neither was entirely satisfactory (September-October 1944). A Napalm soap feeder for use with a continuous mixing device was developed. After the National Foam injection mixer in conjunction with ground Napalm was adopted by the Navy, tests were carried out on specification methods of determining stir time of the ground Napalm (March 1945). The variation in stir time of a representative ground Napalm with Navy aviation gasolines from various sources was determined (April 1945). A few tests were also carried out on agglomeration of ground Napalm at elevated temperatures (May 1945).

Base Mixing.

Near the end of the war base mixing of fuels for use in flame throwers and blaze bombs, for the purpose of improving reliability of performance of these weapons, was considered by the Chemical Warfare Service. Some tests were carried out on variation of consistency with time and temperature for regular and peptized fuels. It was decided that alcohol peptized fuels would be most generally suited for this purpose (August 1945).

Miscellaneous Additives.

During the course of this investigation the effect of a great variety of agents on Napalm fuels was studied. Among these agents were nitrotoluene (March 1944), carbon disulfide (January 1944), motor oil (May 1944), diesel oil (August 1944), zinc and tin (July 1944), polybutene (December 1944), magnesium and other pyrotechnic materials (December 1944), additives for controlling dispersion rate and consistency (January 1945), and miscellaneous chemicals (October 1942). Many agents were found to have a deleterious effect on fuel consistency, including zinc (on long contact), some diesel oils (particularly those dark in color), potassium acetate, etc., in addition to the more common peptizers. When carbon disulfide is substituted for gasoline as the solvent, low temperature ignition of the fuel is greatly improved and considerably greater range can be attained by the flame thrower, providing adequate pressure is supplied.

Cordite Propulsion.

In connection with the investigation of the use of cordite gases as the propelling force in mechanized flame throwers, a study was made of the effect of the combustion products of cordite on thickened fuels. In all cases where the hot combustion gases were fired directly into flame thrower fuel tanks, rapid breakdown of the fuel was found to occur. Most of this breakdown was attributed to the solid and liquid residues carried into the fuel tanks with the gases. No breakdown occurred when the powder gases were brought into contact with the fuel after first being cooled in a receiving vessel. Fuels compounded with excess water ("minimum consistency") and those containing silica gel were found to be somewhat more resistant than regular Napalm to the deleterious effects of the combustion gases (October 1944 to March 1945).

Pumpability of Napalm.

Pumping characteristics of Napalm fuels with various types of pumps were investigated. Capacities of the various pumps for thickened fuels were found to be very nearly the same as for gasoline provided sufficiently large suction piping and pump ports are provided, this requirement being particularly important at consistencies over 300 grams. In connection with this study, a general equation for pressure loss of Napalm fuels in pipes of various sizes was formulated (October, November 1944).

Investigation was also made of ignited jets of thickened fuels propelled by various pumps with a view to using a pump as the propelling force in the mechanized flame thrower. By means of high speed movies and a piezo electric pressure pickup, it was found that the difference between these jets and those propelled by gas pressure was attributable to pulsations in nozzle pressure and jet velocity which occur an integral number of times per revolution of the pump. When these pulsations are too large the jet breaks up into evenly spaced "swarms" of fuel which are incompletely covered with flame. It was found that these pulsations could be completely eliminated by means of a special surge chamber (March, April 1945).

Pump Operated Flame Thrower.

As a result of the investigation of pump propelled jets, an experimental pump operated flame thrower utilizing an Imo screw pump and the E-7 flame gun and mounted on a Buick chassis was assembled. This unit performed so satisfactorily that it was decided to develop a pump operated flame thrower mounted in the M4A3 tank. This project, undertaken shortly before the end of the war, will be completed under Chemical Warfare Service contract (May-August 1945).

Other Thickeners.

From time to time during the course of the investigation, the properties of a variety of thickeners were studied. These included special high gelling power Napalm (August 1944), calcium naphthenate (dilatant) (September 1944), direct reaction aluminum naphthenate (September 1944), substitute (low naphthenic) Napalm (March, May 1945), Valone (December 1942, April 1945), aluminum oresylate (April, May 1945), fatty acids with amines or inorganic hydroxide (November 1944) and Ivory Soap Flakes (September, November 1944). In most cases these studies consisted of laboratory investigations of thickening power, moisture susceptibility, etc. Firing trials were carried out with Valone and soap flakes, performance being poor in both cases.

Fiber Thickeners.

Fibers of nitrocellulose, spun acetate, woodpulp, Saran, polystyrene, newsprint and Vinylite were tried as gasoline thickeners early in the investigation. Of these the pulped newsprint, particularly in conjunction with a small amount of Napalm, appeared most satisfactory. These materials, however, possess a yield value and channel badly in flame thrower fuel tanks. Their performance in tail ejection incendiaries was found to be quite satisfactory. It was found convenient to disintegrate the newsprint in a Wiley Mill or hammer mill prior to dispersion in the gasoline. The most generally satisfactory fiber thickener for the M69 bomb was found to be cellocotton, in the form of wadding or rope. Eleven to fourteen percent of this material in gasoline or gasoline-fuel oil mixtures was found to perform as satisfactorily as the soap thickened fuels in simulated attic structure burning tests (August 1942 to March 1943).

An investigation was made of the feasibility of thickening vesicants with cellulose fibers. Preliminary airplane spray tests with newsprint ground in a laboratory Wiley Mill were quite promising. However, commercially ground samples (Sprout-Waldron) were not as satisfactory. It appeared that the proportion of long and short fibered material was of great importance and that the concentration of the material producing the proper efflux time was very critical. Furthermore, less of the fiber thickened material than of the polymer thickened was in the desired drop-size range (May-September 1943).

Mustard Dispersion Bomb.

The project of developing the M69 bomb for use in spraying mustard was undertaken early in 1943. It was found that when the charge is fired from the bomb without alterations the distribution of mustard is about forty times that required to produce casualties. Numerous devices were tried for causing greater spread. Most satisfactory results were obtained with an inner burster case which is ejected upon impact of the M69. The time

fuse on this inner case is set to go off at a height of 100-250 feet. A number of these bombs were made up and sent to Dugway for testing. Results were sufficiently promising that further development of the weapon was taken over by the Munitions Development Laboratory at the University of Illinois.

Ferro Subcontract.

In the spring of 1944 Ferro Enamel Corp., under subcontract with Eastman Kodak Company, undertook to develop a device whereby Napalm and gasoline could be mixed continuously. By February 1945 a prototype had been assembled and tested by the Chemical Warfare Service. This consisted of a gasoline pump, a hopper feeder which metered the Napalm into the gasoline stream, a gear grinder through which the Napalm-gasoline slurry was forced, a power plant consisting of a small gasoline engine, and a gasoline heater, since the grinder performed satisfactorily only at temperatures of 90° F. or above. Although the unit required considerable modification, it was decided to undertake manufacture of a revised model.

Cleaver Brooks.

The production of the mixers was turned over to the Cleaver-Brooks Company of Milwaukee on purchase order from Eastman Kodak. It was found that after the Ferro mixer had been redesigned along the lines suggested by Chemical Warfare Service, the unit was too heavy and cumbersome to be useful. In the meantime, an alternate form of mixer, consisting of a gasoline engine driven pump for circulating the Napalm-gasoline mix in an open head drum until gelation occurs, had been developed by Cleaver-Brooks. This equipment, designated as the 4-11 mixer, appeared so promising that six units were produced and were being tested by the Armed Forces at the end of the war.

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